

CLAIMS

What is claimed is:

1. A light-weight polyisocyanurate foam composition, comprising:
 - a) an epoxide resin;
 - b) an isocyanate resin;
 - c) a tertiary amine catalyst;
 - d) a cyclic amine.
2. The light-weight polyisocyanurate foam composition of claim 1, wherein the isocyanate resin comprises a mixture of diphenylmethane diisocyanate, methylene bisphenyl isocyanate, and polymethylene polyphenyl isocyanate.
3. The light-weight polyisocyanurate foam composition of claim 1, wherein the epoxide resin is a mixture of either bisphenol A and epichlorohydrin or bisphenol F and epichlorohydrin.
4. The light-weight polyisocyanurate foam composition of claim 3, wherein the quantity of epoxide resin further comprises up to about 50 weight percent of a carboxyl-terminated butadiene acrylonitrile polymer.
5. The light-weight polyisocyanurate foam composition of claim 1, wherein the tertiary amine is 2,4,6-tris(dimethylaminomethyl)phenol.
6. The light-weight polyisocyanurate foam composition of claim 5, wherein the cyclic amine is N,N-dimethylcyclohexylamine.
7. The light-weight polyisocyanurate foam composition of claim 1, wherein said tertiary amine and said cyclic amine are present in a ratio amount equal to about 2:1 to about 2.8:1 of tertiary-to-cyclic amine and wherein the total quantity of amine is present in an amount equal to about 0.5% to about 0.7% of said isocyanate resin.

8. The light-weight polyisocyanurate foam composition of claim 1, wherein the epoxide resin is present in an amount of about 51% to about 56% of said isocyanate resin.
9. The light-weight polyisocyanurate foam composition of claim 1, wherein said epoxide resin and said isocyanate resin are present in a ratio amount of about 1:1.8 to about 1:2 of epoxide resin to isocyanate resin.
10. The light-weight polyisocyanurate foam composition of claim 1, wherein the isocyanate resin is present in an amount of about 60 weight percent to about 65 weight percent of said epoxide resin, said isocyanate resin, and said tertiary and cyclic amines.
11. The light-weight polyisocyanurate foam composition of claim 1, further comprises a bulk filler.
12. The light-weight polyisocyanurate foam composition of claim 11, wherein the bulk filler is present in an amount of about 10 weight percent to about 15 weight percent of said epoxide resin, said isocyanate resin, and said tertiary and cyclic amines.
13. The light-weight polyisocyanurate foam composition of claim 12, wherein the bulk filler is selected from the list consisting of glass microspheres, glass-ceramic cenospheres, multi-cellular glass microspheres, polymeric microspheres, and comminuted form of silicon dioxide, mica, and beta eucryptite, and combinations thereof.
14. The light-weight polyisocyanurate foam composition of claim 13, wherein the bulk filler further comprises fibers selected from the list consisting of fibers such as carbon fibers, e-glass, s-glass, and aramid fibers, and combinations thereof.
15. The light-weight polyisocyanurate foam composition of claim 1, further including a surface active agent.

16. The light-weight polyisocyanurate foam composition of claim 15, wherein the surface active agent is present in an amount of about 2 weight percent to about 5 weight percent of said epoxide resin, said isocyanate resin, and said tertiary and cyclic amines.

17. The light-weight polyisocyanurate foam composition of claim 1, further comprising a quantity of water.

18. The light-weight polyisocyanurate foam composition of claim 17, wherein the water is present in amounts of about 0.05 weight percent to about 0.12 weight percent of said epoxide resin, said isocyanate resin, and said tertiary and cyclic amines.

19. A process for making a high temperature-resistant closed cell polyisocyanurate foam, comprising the steps of:

- a) combining a quantity of an epoxide resin and a quantity of an isocyanate resin to provide a mixed resin mixture;
- b) adding a quantity of a tertiary amine and a quantity of a cyclic amine to resin mixture;
- c) mixing said resin mixture and said tertiary amine and said cyclic amine to provide a pre-expanded foam gel;
- d) dispensing said pre-expanded foam gel into a mold and allowing said gel to react and expand into a closed cell foam;
- e) heating said mold and said expanded foam to about 65°C for about 12 hours to about 16 hours to provide a cured foam member;
- f) cooling said mold and said cured foam member to room temperature;
- g) removing said cured foam member from said mold; and
- h) post-curing said cured foam member by re-heating said cured foam member step-wise to a temperature of about 200°C.

20. The process of claim 19, wherein said isocyanate resin comprises a mixture of diphenylmethane diisocyanate, methylene bisphenyl isocyanate, and polymethylene polyphenyl isocyanate.
21. The process of claim 19, wherein said epoxide resin comprises a mixture of either bisphenol A/epichlorohydrin or bisphenol F and epichlorohydrin.
22. The process of claim 21, wherein the quantity of epoxide resin further comprises up to about 50 weight percent of a carboxyl-terminated butadiene acrylonitrile polymer.
23. The process of claim 19, wherein the tertiary amine is 2,4,6-tris(dimethylaminomethyl)phenol.
24. The process of claim 23, wherein the cyclic amine is N,N-dimethylcyclohexylamine.
25. The process of claim 19, wherein said quantities of said tertiary amine and said cyclic amine are in a ratio amount equal to about 2:1 to about 2.8:1 of tertiary to cyclic amine, and wherein the total quantity of amine is equal to about 0.5% to about 0.7% of said quantity of said isocyanate resin.
26. The process of claim 19, wherein the quantity of epoxide resin is about 51% to about 56% of said quantity of isocyanate resin.
27. The process of claim 21, wherein said epoxide resin and said isocyanate resin are present in a ratio amount of about 1:1.8 to about 1:2 of epoxide resin to isocyanate resin.
28. The process of claim 19, wherein the isocyanate resin is present in an amount of about 60 weight percent to about 65 weight percent of said epoxide resin, said isocyanate resin, and said tertiary and cyclic amines

29. The process of claim 19, wherein the step of combining further comprises the steps of adding a quantity of a bulk filler to said mixed resin mixture, and incorporating said resin mixture and said bulk filler for about 1 minute.
30. The process of claim 29, wherein the bulk filler is present in an amount of about 10 weight percent to about 15 weight percent of said epoxide resin, said isocyanate resin, and said tertiary and cyclic amines.
31. The process of claim 30, wherein the bulk filler is selected from the list consisting of glass microspheres, glass-ceramic cenospheres, multi-cellular glass microspheres, polymeric microspheres, and comminuted form of silicon dioxide, mica, and beta eucryptite, and combinations thereof.
32. The process of claim 31, wherein the bulk filler further comprises fibers selected from the list consisting of fibers such as carbon fibers, e-glass, s-glass, and aramid fibers, and combinations thereof.
33. The process of claim 19, wherein said step of combining said epoxide resin and said isocyanate resin further includes adding a quantity of a surface active agent.
34. The process of claim 33, wherein the surface active agent is present in an amount of about 2 weight percent to about 5 weight percent of said epoxide resin, said isocyanate resin, and said tertiary and cyclic amines.
35. The process of claim 19, wherein the step of mixing said resin mixture and said tertiary and cyclic amines further comprises the step of adding a quantity of water.
36. The process of claim 35, wherein the water is present in amounts of about 0.05 weight percent to about 0.12 weight percent of said epoxide resin, said isocyanate resin, and said tertiary and cyclic amines.
37. The process of claim 19, wherein the step of post-curing further comprises the step of increasing the temperature of said cured foam member to 200°C over a period of at least about 36 hours.

38. The process of claim 37, wherein the step of increasing said temperature further comprises the steps of:

- a) heating the cured foam member to the 65°C for about 2 hours;
- b) increasing the temperature of the cured foam member up to 150°C over about 8 hours and holding the temperature of the cured foam member at about to 150°C for an additional 5 hour;
- c) increasing the temperature of the cured foam member to about 180°C over about 8 hours and holding the temperature of the cured foam member at about to 180°C for an additional 5 hours;
- d) increasing the temperature of the cured foam member to about 200°C over about 5 hours and holding the temperature of the cured foam member at about to 200°C for an additional 5 hour;
- e) deceasing the temperature of the cured foam member about to 65°C over about 5 hours and holding the temperature of the cured foam member at about to 65°C for an additional 1 hour; and
- f) cooling the temperature of the cured foam member to room temperature.

39. A tool made by a process comprising the steps of:

- a) combining an epoxide resin and an isocyanate resin to provide a resin mixture;
- b) adding a mixture of an tertiary amine and a cyclic amine to said resin mixture, said amine initiating a reaction between said epoxide resin and said isocyanate resin to form a polymer gel;
- c) dispensing said polymer gel into a mold;
- d) curing the polymer gel at a temperature of about 65°C to provide a polyisocyanurate foam member;
- e) cooling said mold and de-molding said polyisocyanurate foam member;
- f) post-curing the polyisocyanurate foam member by re-heating the polyisocyanurate foam member step-wise to a temperature of about 200°C;

- g) cooling said post-cured polyisocyanurate foam member to room temperature to provide a post-cured polyisocyanurate foam tool, said post-cured foam tool sustaining prolonged exposure to temperatures up to about 200°C while also maintaining a mechanical compressive strength of at least 25 MPa.
40. The tool of claim 39, wherein said isocyanate resin comprises a mixture of diphenylmethane diisocyanate, methylene bisphenyl isocyanate, and polymethylene polyphenyl isocyanate.
41. The tool of claim 39, wherein said epoxide resin comprising a mixture of bisphenol A and epichlorohydrin resins or a mixture of bisphenol F and epichlorohydrin resins.
42. The tool of claim 41, wherein the quantity of epoxide resin further comprises up to about 50 weight percent of a carboxyl-terminated butadiene acrylonitrile polymer.
43. The tool of claim 39, wherein the tertiary amine is 2,4,6-tris(dimethylaminomethyl)phenol.
44. The tool of claim 43, wherein the cyclic amine is N,N-dimethylcyclohexylamine.
45. The tool of claim 39, wherein said quantities of said tertiary amine and said cyclic amine are in a ratio amount equal to about 2:1 to about 2.8:1 of tertiary to cyclic amine, and wherein the total quantity of amine is equal to about 0.5% to about 0.7% of said quantity of said isocyanate resin.
46. The tool of claim 39, wherein the quantity of epoxide resin is about 51% to about 56% of said quantity of isocyanate resin.
47. The tool of claim 39, wherein said epoxide resin and said isocyanate resin are present in a ratio amount of about 1:1.8 to about 1:2 of epoxide resin to isocyanate resin.

48. The tool of claim 39, wherein the isocyanate resin is present in an amount of about 60 weight percent to about 65 weight percent of said epoxide resin, said isocyanate resin, and said tertiary and cyclic amines.
49. The tool of claim 39, wherein said step of combining said epoxide resin and said isocyanate resin further includes adding a quantity of a surface active agent.
50. The tool of claim 49, wherein the surface active agent is present in an amount of about 2 weight percent to about 5 weight percent of said epoxide resin, said isocyanate resin, and said tertiary and cyclic amines.
51. The tool of claim 39, wherein the step of combining further comprises the steps of adding a quantity of a bulk filler to said mixed resin mixture, and incorporating said resin mixture and said bulk filler for about 1 minute.
52. The tool of claim 51, wherein the bulk filler is present in an amount of about 10 weight percent to about 15 weight percent of said epoxide resin, said isocyanate resin, and said tertiary and cyclic amines.
53. The tool of claim 52, wherein the bulk filler is selected from the list consisting of glass microspheres, glass-ceramic cenospheres, multi-cellular glass microspheres, polymeric microspheres, and comminuted form of silicon dioxide, mica, and beta eucryptite, and combinations thereof.
54. The tool of claim 53, wherein the bulk filler further comprises fibers selected from the list consisting of fibers such as carbon fibers, e-glass, s-glass, and aramid fibers, and combinations thereof.
55. The tool of claim 51, wherein the bulk filler is present in an amount of about 10 weight percent to about 15 weight percent of said epoxide resin, said isocyanate resin, and said tertiary and cyclic amines.
56. The tool of claim 39, wherein said step of combining said epoxide resin and said isocyanate resin further includes adding a quantity of water.

57. The tool of claim 39, wherein the step of post-curing further comprises the step of increasing the temperature of said cured foam member to 200°C over a period of at least about 36 hours.

58. The tool of claim 57, wherein the step of increasing said temperature further comprises the steps of:

- a) heating the cured foam member to the 65°C for about 2 hours;
- b) increasing the temperature of the cured foam member up to 150°C over about 8 hours and holding the temperature of the cured foam member at about to 150°C for an additional 5 hours;
- c) increasing the temperature of the cured foam member to about 180°C over about 8 hours and holding the temperature of the cured foam member at about to 180°C for an additional 5 hours;
- d) increasing the temperature of the cured foam member to about 200°C over about 5 hours and holding the temperature of the cured foam member at about to 200°C for an additional 5 hours;
- e) decreasing the temperature of the cured foam member about to 65°C over about 5 hours and holding the temperature of the cured foam member at about to 65°C for an additional 1 hour; and
- f) cooling the temperature of the cured foam member to room temperature.

59. The tool of claim 58, further including the step of milling or shaping one or more surfaces to provide a 3-dimensional tool contour.

60. The tool of claim 59, further including coating the 3-dimensional tool contour with a polymer conformal coating.

61. The tool of claim 59, further comprising a positive or negative relief image on a surface and wherein said tool is adapted for use as a hot embossing tool master.

62. The tool of claim 59, further comprising one or more contiguous inserts forming a positive or negative 3-dimensional image, and wherein said one or more contiguous inserts are adapted for use as an injection mold die tool.

63. A large tool assembly comprising the step of gluing or bonding together two or more individual tools prepared by the process of claim 59.

64. A tool made by a process comprising the steps of:

- a) combining an epoxide resin and an isocyanate resin to provide a resin mixture;
- b) adding a mixture of an tertiary amine and a cyclic amine to said resin mixture, said amine initiating a reaction between said epoxide resin and said isocyanate resin to form a polymer gel;
- c) applying said polymer gel onto a surface;
- d) curing the polymer gel at a temperature of about 65°C to provide a polyisocyanurate foam layer;
- e) cooling said polyisocyanurate foam layer;
- f) post-curing the polyisocyanurate foam layer by re-heating the polyisocyanurate foam member step-wise to a temperature of about 200°C;
- g) cooling said post-cured polyisocyanurate foam layer to room temperature, to provide a foam tool for sustaining prolonged exposure to temperatures up to about 200°C while also maintaining a mechanical compressive strength of at least 25 MPa.

65. The tool of claim 64, wherein the step of applying further comprises spraying said foam gel onto said surface.

66. A high temperature insulation medium comprising the composition of claim 12.

67. A high temperature insulation medium made by the process of claim 30.